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AN APPARATUS FOR FUMIGATING INSECTS WITH HYDROCYANIC ACID GAS IN THE LABORATORY

By E. M. Livingstone,
Division of Control Investigations 1/

In studying the toxicity of hydrocyanic acid gas to the larva of the cigarette beetle (Lasioderma serricorne (F.)) the apparatus here described was assembled. No originality in its design is claimed, for it embodies features contained in apparatus described by a number of investigators who have worked with various fumigants. The purpose of this paper is to describe the assembled apparatus, giving suggestions for its economical construction and practical operation.

APPARATUS

The apparatus consists of a flowmeter, hydrocyanic acid gas generators, fumatorium, absorption bottles, and suction apparatus. These are connected with glass tubing in the order named in such a way that air enters the flowmeter, where its volume may be determined, passes next through the generators where hydrocyanic acid gas is taken up, then to the fumatorium containing the insect material, then through the absorption bottles where the fumigant is absorbed, and finally to the suction apparatus. Figure 1 is a diagram showing the relationship of these units.

Flowmeter

The flowmeter, manometer tube type, is used to measure the quantity of air passing through the apparatus in any given period. The flowmeter used in this apparatus is shown in figure 2. Air passing through a horizontal glass tube, a small portion of which is constricted, causes a differential in pressure before and after the constriction. This differential is measured by attaching one end of a vertical U-tube, which has previously been filled with a fluid, in this case heptane, to the horizontal tube before the

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constriction and the other end after it. The pressure differential results in a variation between the levels of the heptane in the two arms of the U-tube, and this variation is proportional to the rate of air flow and hence serves as a gauge of the quantity of air that passes through the constricted tube in any given length of time.

It is obvious, therefore, that the size of the opening in the constricted tube and the force of suction applied to the tube affect the rate of air flow. In addition there are other minor factors that affect the rate of flow, or the variation in the levels of the heptane in the U-tube, and hence it is necessary to calibrate each meter of this type in its actual position in the apparatus of which it is a part.

The flowmeter shown in figure 2 was made up in the laboratory from two T-tubes; two No. 1, 1-hole, rubber stoppers; a piece of capillary tubing about 1 inch in length (actually in this case a piece of the stem of a broken thermometer); a section of glass tubing of approximately the same diameter as that of the T-tubes; and two short pieces of tubing for the purpose of making connections. Details of construction and the use of these materials will not be set down, as it is believed that the drawing shown in figure 2 is adequate for directions.

Generators and Trap Bottle

The hydrocyanic acid gas generators, the needle valve for controlling the rate of air flow, and the trap bottle for collecting any spray that may pass out of the generators with the air-gas mixture are illustrated in figure 3. The intake end of the needle valve is attached directly to the outlet of the flowmeter with a rubber tube connection. The exit end is attached to the first of the series of three generators. The needle valve should be capable of micro-adjustment.

The generators consist of three wide-mouthed 500-cc. bottles, connected in series so that a stream of air may be drawn through them, each containing a mixture of hydrochloric acid, sodium cyanide, and water capable of liberating the desired amount of hydrocyanic acid gas into the air stream. A large excess of hydrochloric acid is to be avoided. For each milligram of HCN required per liter of air, 0.1 cc. of concentrated hydrochloric acid and 0.057 gram of sodium cyanide (96 percent) are added to 100 cc. of distilled water. The amounts of hydrochloric acid and sodium cyanide should be regarded as only close approximations; the exact amount of each will vary with each lot of acid and cyanide. The desired concentration may be readily obtained by making up small quantities of solutions having the proportions of acid and cyanide in different amounts for use in preliminary analyses, and from the results of these the right amount of acid

and cyanide to use to get the wanted concentration may be calculated or interpolated. Care should be taken to have the air-intake tubes in each bottle extend nearly to the bottom of the solution; outlet tubes should be well above the surface of the liquid to guard against spray entering them. To prevent spray from entering the fumatorium, a trap bottle, similar to those used to hold the sodium cyanide solution but empty, is placed between the last generator and the fumatorium.

Fumatorium

From the trap bottle the mixture of air and HCN passes into the fumatorium. (When the apparatus is immersed in a thermo-regulated water bath, the gaseous mixture may be passed through a coil of glass tubing to insure its reaching the temperature of the bath before it enters the fumatorium.) As shown in figure 4, the fumatorium is composed of five tubes, 1 inch in diameter and 6 inches in length, joined in series by having the exhaust of the first lead to the intake opening of the next, and so on. The gas enters at the top of the tubes, flows downward over the insects, and passes out near the bottom. Each tube can be isolated from the rest for the removal of insects by closing the proper stopcocks located in the connecting tubes. Thus with five tubes joined in series, as many (i. e. five) different exposures may be made without diluting the gas mixture, for the insects may be removed from a tube after first isolating it from the adjacent tubes. The first lot of insects removed must be taken from the last tube in the series, the second from the next to last, and so on. The removal of insects from a tube interrupts the flow of the gas stream momentarily, but as soon as the insects are removed, the tube is closed, and the stopcocks are opened again, the flow proceeds. Such a short interruption in flow did not seriously affect the results of experiments under way.

Another type of fumatorium, differing from the one described in design but not in principle, is made up of five U-shaped drying tubes. The intakes and exits of the tubes are welded together in a manner to permit the air-gas mixture to flow from the first to the last. Stopcock glass stoppers are an added improvement to this type of fumatorium, since rubber stoppers, such as those used in the fumatorium described above, "sorb" small amounts of hydrocyanic acid gas. The glass stoppers are used to isolate individual tubes, thus obviating the necessity of having stopcocks in the tubes connecting the individual chambers of the fumatorium.

Absorption Bottles

The air-gas mixture, after leaving the fumatorium, passes into one of two absorption bottles (fig. 5), each of which contains a 5-percent solution of sodium hydroxide. The two bottles are connected to the exit of the fumatorium with tubes having 3-way

stopcocks. The exits from the bottles are connected to the suction apparatus by similar means. It is thus possible to direct the stream of gas through either bottle desired. One bottle is used for taking or absorbing samples of gas for analysis; at all times when sampling is not in progress the flow is directed through the other bottle. Thus all hydrocyanic acid gas is absorbed from the stream, and only air enters the suction apparatus.

Suction Apparatus

A diagram of the suction apparatus is shown in figure 6. The siphon bottle is connected directly to the exit tubes of the absorption bottles. It should be emphasized, in setting up the suction apparatus, that any backward flow of either air or water in the siphon tube at any time should be prevented. Should this happen, as when the tube is jarred or suddenly lifted from the reservoir bottle into which the water is siphoning, air will flow backward into the absorption bottles, causing the sodium hydroxide solution in them to be forced over into the fumatorium. This occurrence may be prevented by allowing the siphon tube to terminate under water after having made a loop, the bottom of which extends below the level of the water. For this purpose, as shown in the diagram, the siphon tube terminates in a 1-liter distilling flask, after having been looped lower than the exit tube of the distilling flask. The water coming from the exit tube of the flask may be caught readily in a measuring cylinder during sampling, or in a large bottle at other times. In this way, the siphon tube is not disturbed during sampling.

Calibration of Flowmeter

The flowmeter may be calibrated after the complete apparatus is assembled, and for this purpose the cyanide solution and the sodium hydroxide solution may be omitted from their respective bottles and water substituted in them. The flowmeter is calibrated by measuring the volume of air (approximately equivalent to the quantity of water displaced in the siphon bottle) during stated intervals, when the difference in the levels of the heptane in the U-tube is held constant. The difference is controlled by the needle valve between the flowmeter and the generators and may be measured with a small millimeter rule, or, more conveniently, by attaching a rectangular piece of stiff millimeter paper behind the arms of the U-tube. This paper should be slightly wider than the horizontal distance between the arms of the U-tube and as long as the tube. It may be fastened to the back of the U-tube with rubber bands, as such an attachment permits its being slipped easily up or down the arms, thus making it unnecessary to adjust the level of the liquid to the zero of the scale before an experiment is begun, as would be required were the scale mounted in a fixed position.

In the experimental work the calibrated flowmeter is used to obtain the flow of air desired. As a further check on its continued accuracy during periods of use, occasionally in taking samples of gas for analysis the amount of water displaced during the sampling period is actually caught in the measuring cylinder, as shown in figure 6, and its volume compared with the amount indicated by the flowmeter. Should a discrepancy occur, the flowmeter should be carefully cleaned and rechecked until the readings are accurate. Although the volume of water displaced is theoretically not exactly equivalent to the volume of gas passing through the flowmeter, the difference between the two is small and in practical work may be disregarded.

Samples of gas are taken for analysis at intervals during the fumigation procedure, usually immediately before the removal of a test lot of insects from the fumatorium, and at intervals not greater than 1 hour apart. With most concentrations, the length of a sampling period is about 15 minutes, during which time the rate of flow should be about 30 cc. per minute. At the beginning of a fumigation the apparatus should be flushed out, after the insects are inserted, by increasing the rate of flow for about 1 minute.

GAS ANALYSIS

The concentration of hydrocyanic acid gas is determined by the Liebig method.2/ By passing a known quantity of the gas-air mixture through a 5-percent solution of sodium hydroxide, the gas is converted to sodium cyanide, which, immediately as it forms, dissolves in the water of the solution. The following equation represents this reaction:

$$NaOH + HCN = NaCN + H_2O.$$

To this solution a few drops of 20-percent potassium iodide solution are added to serve as an indicator. Instead of adding the iodide salt in solution, a few crystals of the salt dropped into the sodium cyanide solution are equally as satisfactory for titration purposes. The whole is then titrated with N/50 silver nitrate solution, which by its reaction with sodium cyanide forms a complex salt, sodium silver cyanide. The silver nitrate is added very slowly by small drops, and after each drop the solution is gently shaken. With the addition of each drop the solution momentarily takes on a slight turbidity, but, until all of the sodium cyanide has been converted, this turbidity will disappear on shaking. When all the sodium cyanide has been converted to this salt, any further addition of silver nitrate reacts with the potassium indicator to form silver iodide, which, being insoluble, gives a

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2/ Scott, W. W. 1925. Standard Methods of Chemical Analysis. Ed. 4, illus. New York.

permanent, turbid precipitate, the appearance of which indicates that the end point of the titration has been reached. The titration is best when carried out in a darkened room, where, by means of a beam of artificial light directed on the solution, the first appearance of the permanent precipitate is easily observed. The reactions involved in the titration are as follows:

$$2NaCN + AgNO_3 = NaAg(CN)_2 + NaNO_3$$

$$KI + AgNO_3 = KNO_3 + AgI.$$

From the data obtained in the analysis the concentration of hydrocyanic acid gas may be calculated as follows:

$$\frac{1.08 \times (\text{Number cc. of } AgNO_3)}{\text{Volume gas sample in liters}} = \text{Mg. of HCN per liter of air.}$$

Dosages expressed in milligrams per liter may be stated in terms of ounces per 1,000 cubic feet by use of the equation

1 mg. per liter = 1 oz. per 1,000 cu. ft.

MISCELLANEOUS

To secure temperature control the apparatus may be immersed in a thermo-regulated water bath.3/

Care should be taken to see that all apparatus is thoroughly cleansed, rinsed with distilled water, and dried before use, as the presence of water or alkali in the connecting tubes and fumatorium will cause errors in concentration by sorbing the gas. Reduce rubber connections to a minimum where these will be contacted by the stream of air-gas mixture, as rubber takes up hydrocyanic acid gas. The ends of glass tubing preferably should be ground and pushed together as closely as possible within the rubber connections. Rubber stoppers may be coated lightly with vaseline to prevent sorption. In order to reduce errors that may be caused by sorption of the gas by rubber and the glass walls, the apparatus should be operated for about an hour previous to the start of actual experimentation to allow their sorptive capacities to become satisfied to a large degree. After this period the insect material may be placed in the fumatorium.

CAUTION: Hydrocyanic acid is a deadly poison, and this fact should be ever kept in mind when working in the laboratory with this apparatus.

3/ Davis, L. H., and Livingstone, E. M. 1937. A Small Thermo-Regulated Water Bath Heater. U. S. Dept. Agr., Bur. Ent. and Plant Quar., Circular ET-98 (multigraphed).

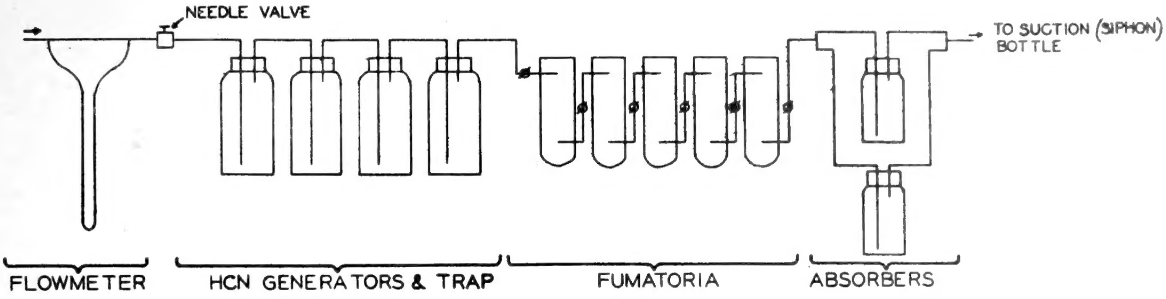

Figure 1.--Diagram showing the arrangement of the component parts of the fumigation apparatus.

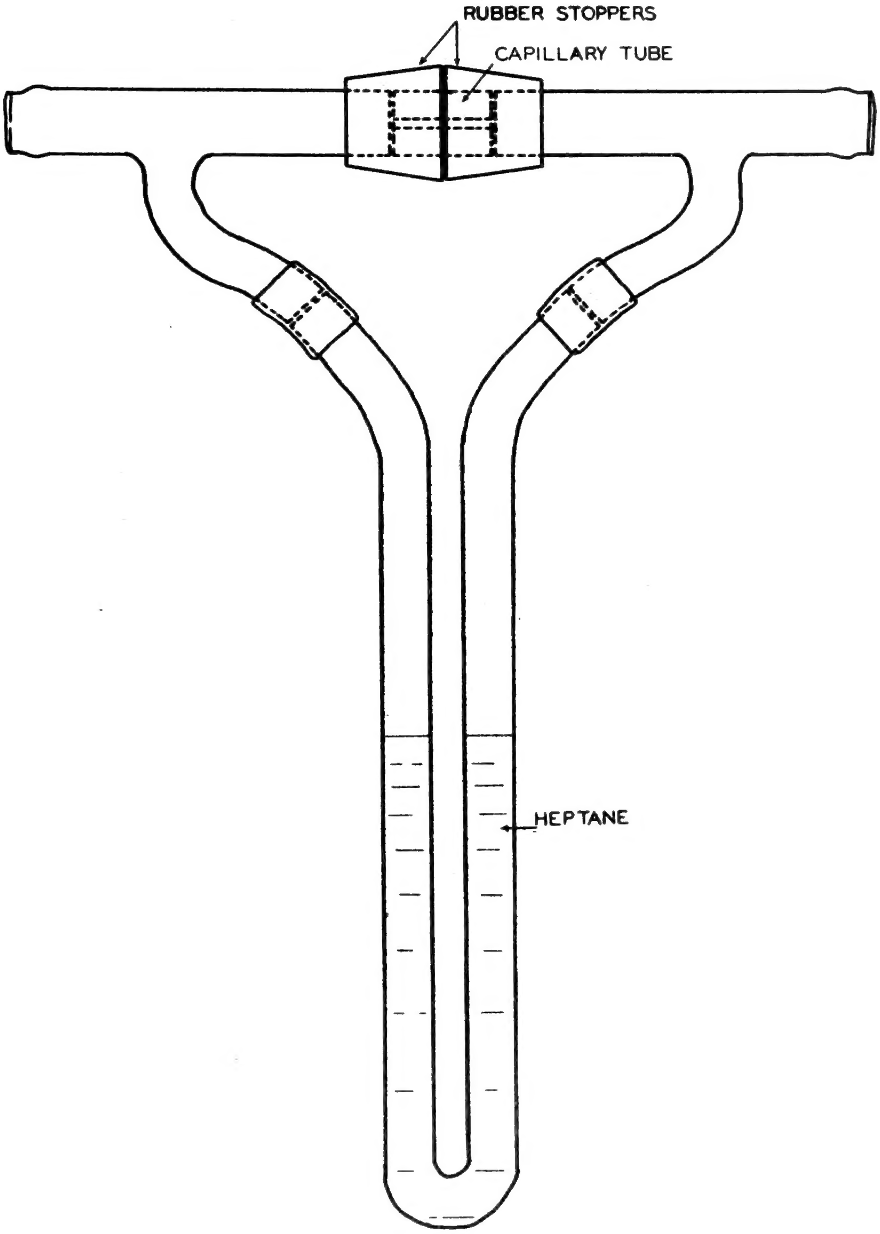

Figure 2.--Diagram showing the type of manometer tube used as flowmeter in the fumigation apparatus.

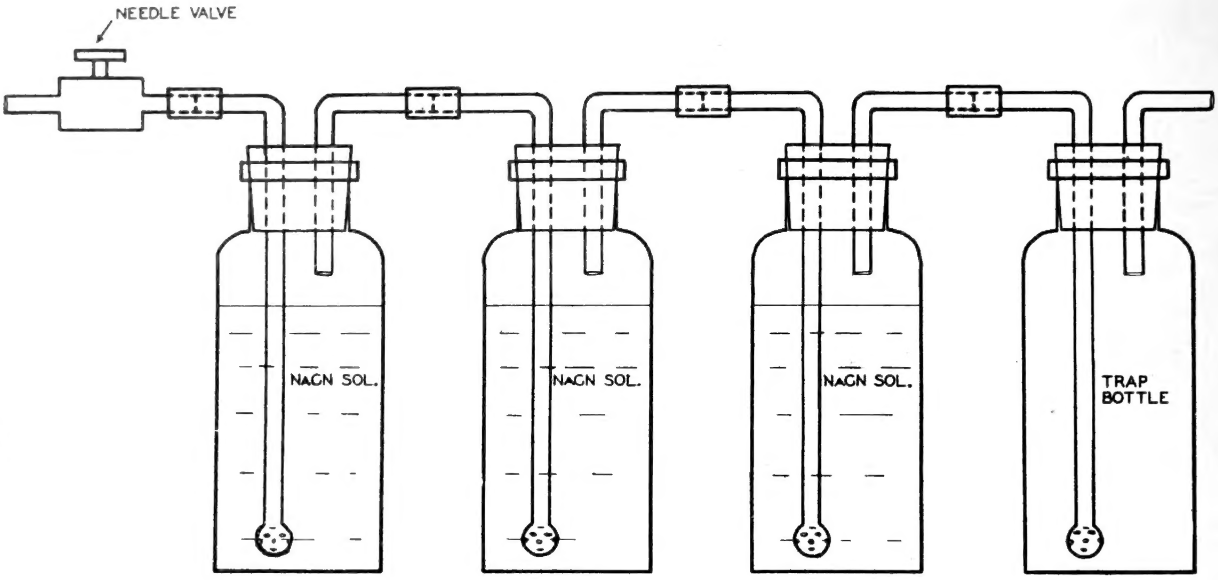

Figure 3.--Diagram showing needle valve (for controlling rate of air-flow), hydrocyanic acid gas generators, and trap bottle.

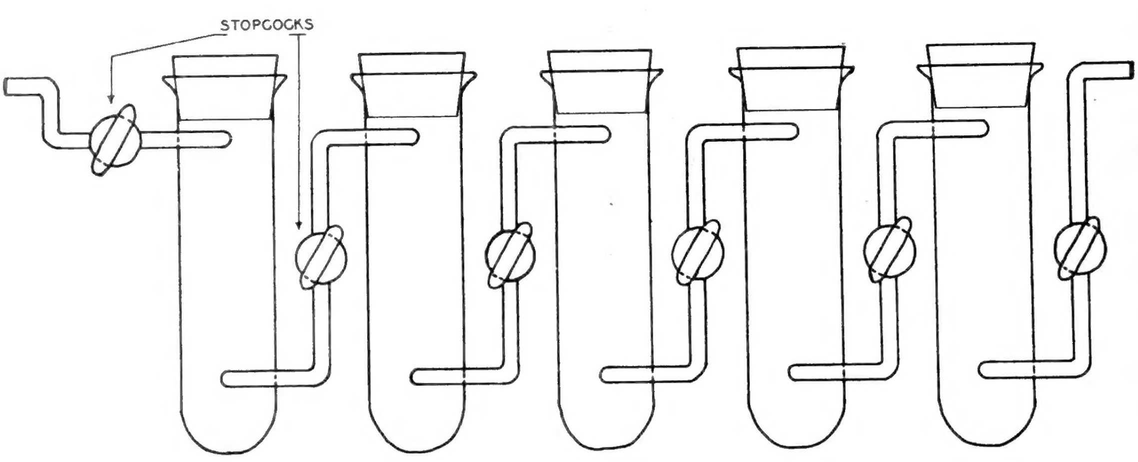

Figure 4.--Diagram showing arrangement of 5 test tubes connected in series to serve as a fumatorium.

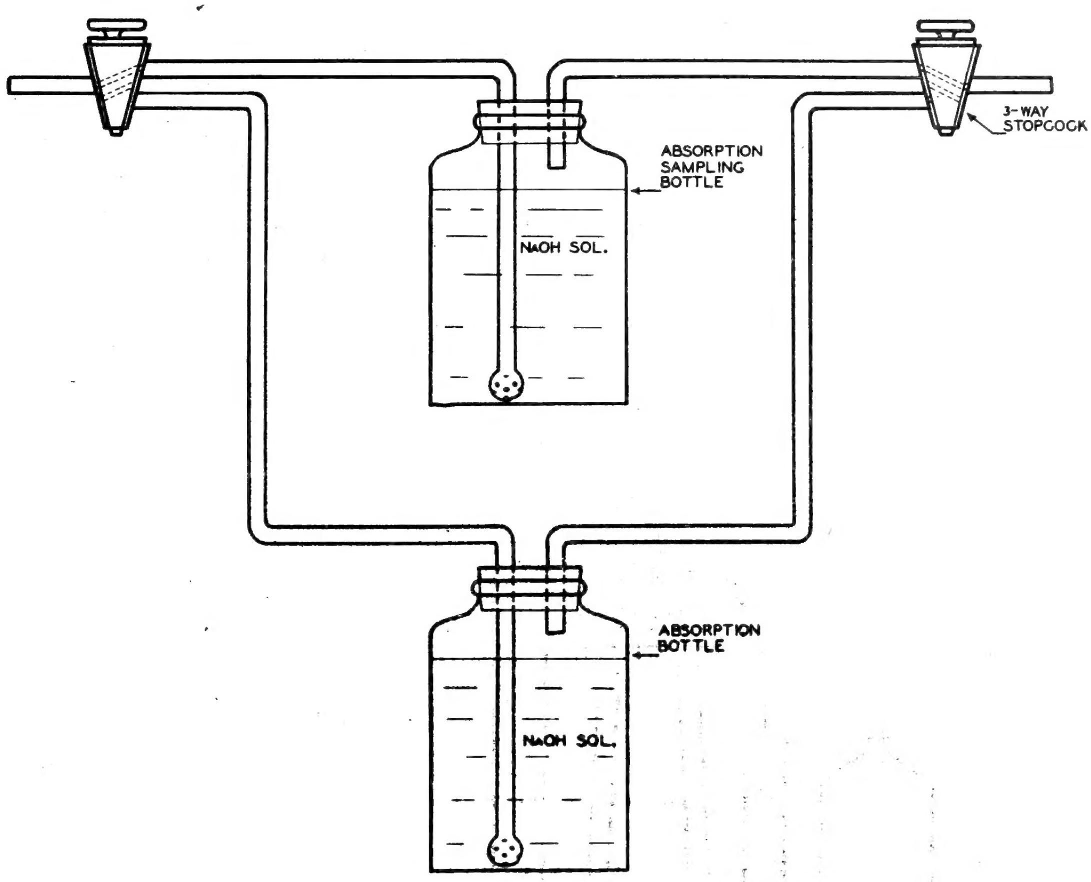

Figure 5.—Diagram showing gas absorption and sampling bottles.

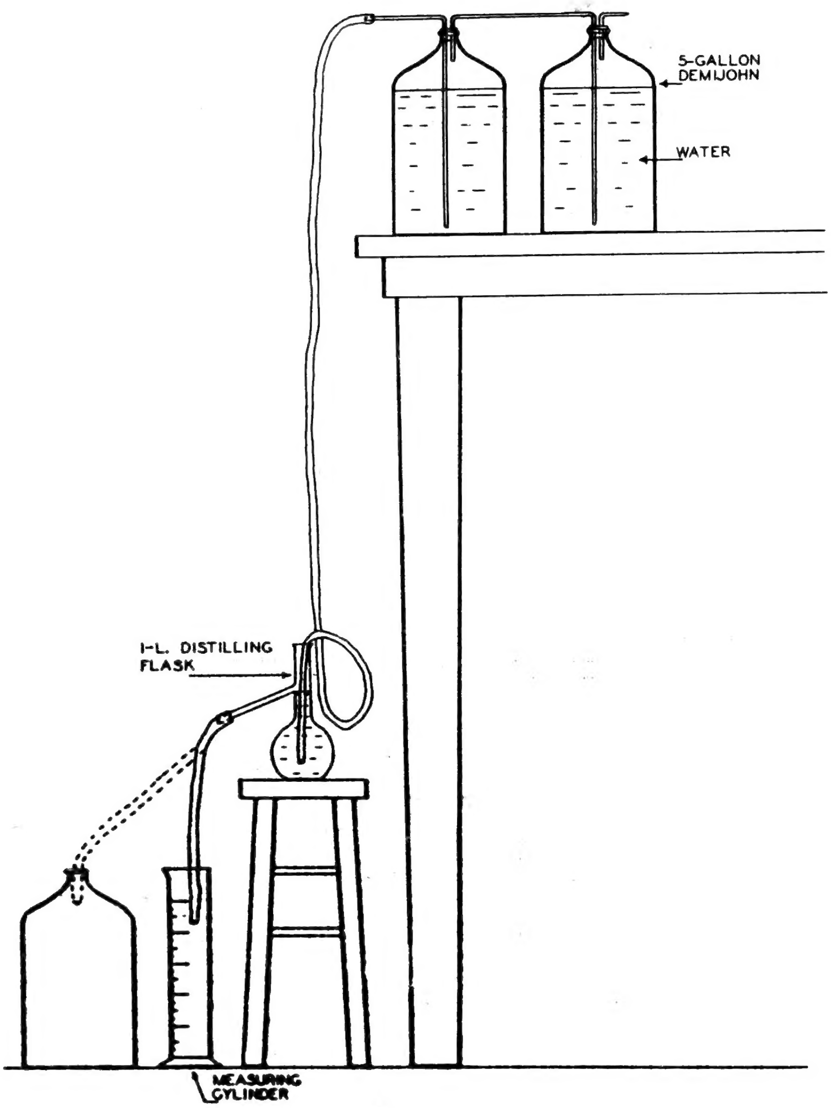

Figure 6.--Diagram of arrangement of suction-creating apparatus.